भारतीय मानक
Indian Standard

IS 3025 (Part 19) : 2018

जल एवं अपशिष्ट जल के नमूने लेने तथा परीक्षण (भौतिक एवं रसायन) की पद्धतियाँ

भाग 19 पृथकृत पदार्थ

(दूसरा पुनरीक्षण)

Methods of Sampling and Test (Physical and Chemical) for Water and Waste Water

Part 19 Settleable Matter

(Second Revision)

ICS 13.060.50

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July 2018

Price Group 2

Water Quality Sectional Committee, CHD 36

FOREWORD

This Indian Standard (Second Revision) was adopted by the Bureau of Indian Standards, after the draft finalized by the Water Quality Sectional Committee had been approved by the Chemical Division Council.

This standard was first revised in 1984 and it includes two methods for the determination of settleable matter gravimetric method and volumetric method.

Solids refer to matter suspended or dissolved in water or waste water. Solids may affect water or effluent quality adversely in a number of ways. Waters with high dissolved solids generally are of inferior palatability and may induce an unfavourable physiological reaction in the transient consumer. For these reasons, a limit of 500 mg dissolved solids / litre is desirable for drinking waters. Highly mineralized waters are also unsuitable for many industrial applications. Waters high in suspended solids may be aesthetically unsatisfactory for use as they would appear muddy/turbid.

Solids analyses are important in the control of biological and physical wastewater treatment processes and for assessing compliance with regulatory agency wastewater effluent limitations. This standard deals with the determination of suspended solids in water and wastewater. In this revision, both the methods have been revised with considerable assistance from APHA 2540-D and APHA 2540-F.

In reporting the results of a test or analysis in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 ‘Rules for rounding off numerical values (*revised*)’.

Indian Standard

METHODS OF SAMPLING AND TEST (PHYSICAL AND CHEMICAL) FOR WATER AND WASTE WATER

PART 19 SETTLEABLE MATTER

(Second Revision)

1 SCOPE

This standard prescribes two methods, one gravimetric and the other volumetric, for the determination of settleable matter. These methods are applicable to all types of water and waste water.

2 REFERENCES

The following standards contain provisions which, through reference in this text constitute provisions of this standard. At the time of publications, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below.

IS No.	*Title*
1070 : 1992	Reagent grade water
3025	Methods of sampling and test (physical and chemical) for water and wastewater :
(Part 15) : 1984/ ISO 7888	Total residue (total solids — dissolved and suspended)
(Part 16) : 1984	Filterable residue (total dissolved solids)

3 GRAVIMETRIC METHOD

3.1 Principle

A well-mixed sample is filtered through a weighed standard glass-fiber filter and the residue retained on the filter is dried at 103–105°C and weighed till a constant weight is achieved. The increase in weight of the filter represents the total suspended solids. If the suspended material clogs the filter and prolongs filtration, it may be necessary to increase the diameter of the filter or decrease the sample volume. To obtain an estimate of total suspended solids, calculate the difference between total dissolved solids [IS 3025 (Part 16)] and total solids [IS 3025 (Part 15)].

3.2 Interferences

Exclude large floating particles or submerged agglomerates of non-homogenous materials from the sample if it is determined that their inclusion is not representative. Because excessive residue on the filter may form a water-entrapping crust, limit the sample size to that yielding no more than 200 mg residue. For samples high in dissolved solids thoroughly wash the filter to ensure removal of dissolved material. Prolonged filtration times resulting from filter clogging may produce high results owing to increased colloidal materials captured on the clogged filter.

3.3 Apparatus

3.3.1 *Desiccator*, provided with a desiccant containing a colour indicator of moisture concentration or an instrumental indicator.

3.3.2 *Drying Oven*, for operation at 103 to 105°C.

3.3.3 *Analytical Balance*, capable of weighing to 0.1 mg.

3.3.4 *Magnetic Stirrer*, with TFE stirring bar.

3.3.5 *Wide-bore Pipets*

3.3.6 *Graduated Cylinder*, capacity 1 litre.

3.3.7 *Low-form beaker*

3.3.8 *Glass-fiber Filter Disks*, without organic binder.

3.3.9 *Filtration Apparatus* — One of the following, suitable for the filter disk selected:

a) Membrane filter funnel;
b) Gooch crucible, 25-ml to 40-ml capacity, with Gooch crucible adapter; or
c) Filtration apparatus with reservoir and coarse (40 to 60 µm) fritted disk as filter support.

3.3.10 *Suction Flask*, of sufficient capacity for sample size selected.

3.3.11 *Aluminium Weighing Dishes*

3.4 Procedure for Determination of Total Suspended Solids

3.4.1 *Preparation of Glass-fiber Filter Disk*

If pre-prepared glass-fiber filter disks are used, eliminate this step. Insert disk with wrinkled side up in filtration apparatus. Apply vacuum and wash disk with three successive 20-ml portions of reagent-grade water (*see* IS 1070). Continue suction to remove all traces of water, turn vacuum off, and discard washings. Remove filter from filtration apparatus and transfer to an inert aluminium weighing dish. If a Gooch crucible is used, remove crucible and filter combination.

Dry in an oven at 103 to 105°C for 1 h. Cool in desiccator to balance temperature and weigh. Repeat cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until weight change is less than 4 percent of the previous weighing or 0.5 mg, whichever is less. Store in desiccator until needed.

3.4.2 *Selection of Filter and Sample Sizes*

Choose sample volume to yield between 2.5 and 200 mg dried residue. If volume filtered fails to meet minimum yield, increase sample volume up to 1 litre. If complete filtration takes more than 10 min, increase filter diameter or decrease sample volume.

3.4.3 *Sample Analysis*

Assemble filtering apparatus and filter and begin suction. Wet filter with a small volume of reagent-grade water to seat it. Stir sample with a magnetic stirrer at a speed to shear larger particles, if practical, to obtain a more uniform (preferably homogeneous) particle size. Centrifugal force may separate particles by size and density, resulting in poor precision when point of sample withdrawn is varied. While stirring, pipet a measured volume onto the seated glass-fiber filter. For homogeneous samples, pipet from the approximate midpoint of container but not in vortex. Choose a point both middepth and midway between wall and vortex. Wash filter with three successive 10-ml volumes of reagent grade water, allowing complete drainage between washings, and continue suction for about 3 min after filtration is complete. Samples with high dissolved solids may require additional washings. Carefully remove filter from filtration apparatus and transfer to an aluminium weighing dish as a support.

Alternatively, remove the crucible and filter combination from the crucible adapter if a Gooch crucible is used. Dry for at least 1 h at 103 to 105 °C in an oven, cool in a desiccator to balance temperature, and weigh. Repeat the cycle of drying, cooling, desiccating, and weighing until a constant weight is obtained or until the weight change is less than 4 percent of the previous weight or 0.5 mg, whichever is less.

Analyze at least 10 percent of all samples in duplicate. Duplicate determinations should agree within 5 percent of their average weight.

3.4.4 *Calculation of Suspended Solids*

$$\text{mg of suspended solids/l} = \frac{(A - B) \times 1000}{\text{Sample volume, ml}}$$

where

A = weight of filter + dried residue, mg, and

B = weight of filter, mg.

3.4.5 *Precision*

The standard deviation was 5.2 mg/l (coefficient of variation 33%) at 15 mg/l, 24 mg/l (10%) at 242 mg/l, and 13 mg/l (0.76%) at 1 707 mg/l in studies by two analysts of four sets of 10 determinations each.

Single-laboratory duplicate analyses of 50 samples of water and wastewater were made with a standard deviation of differences of 2.8 mg/l.

3.5 Procedure for Determination of Non-Settleable Solids

3.5.1 Pour a well-mixed sample into a glass vessel of not less than 9 cm diameter, a quantity not less than 1 litre and sufficient to give a depth of 20 cm. Alternatively, use a glass vessel of greater diameter and a larger volume of sample. Let stand quiescent for 1 h and without disturbing the settled or floating material, siphon 250 ml from centre of container at a point halfway between the surface of the settled material and the liquid surface. Determine total suspended solids (mg/l) of this supernatant liquid using procedure given at **3.4**. These are the non-settleable solids.

3.6 Calculation of Settleable Solids

mg settleable solids/l = mg total suspended solids/l – mg non-settleable solids/l

4 VOLUMETRIC METHOD

4.1 Principle

Volume of settleable matter is determined by allowing the sample to stand for 1 h in an Imhoff cone.

4.2 Apparatus

Standard Imhoff cone, suitably calibrated bottom to top and having 1-litre mark.

4.3 Procedure

4.3.1 Fill an Imhoff cone to the 1-litre mark with a well-mixed sample. Settle for 45 min, gently agitate sample near the sides of the cone with a rod or by spinning, settle 15 min longer, and record volume of settleable solids in the cone as milliliters per liter. If the settled matter contains pockets of liquid between large settled particles, estimate volume of these and subtract from volume of settled solids. The practical lower limit of measurement depends on sample composition and generally is in the range of 0.1 to 1.0 ml/l. Where a separation of settleable and floating materials occurs, do not estimate the floating material as settleable matter.

4.3.2 Where biological or chemical floc is present, the gravimetric method is preferred, and the procedures given at **3.4**, **3.5** and **3.6** is followed.

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This Indian Standard has been developed from Doc No.: CHD 36 (11524).

Amendments Issued Since Publication

Amend No.	Date of Issue	Text Affected

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Published by BIS, New Delhi